CEBAF PR-93-020
June 1993

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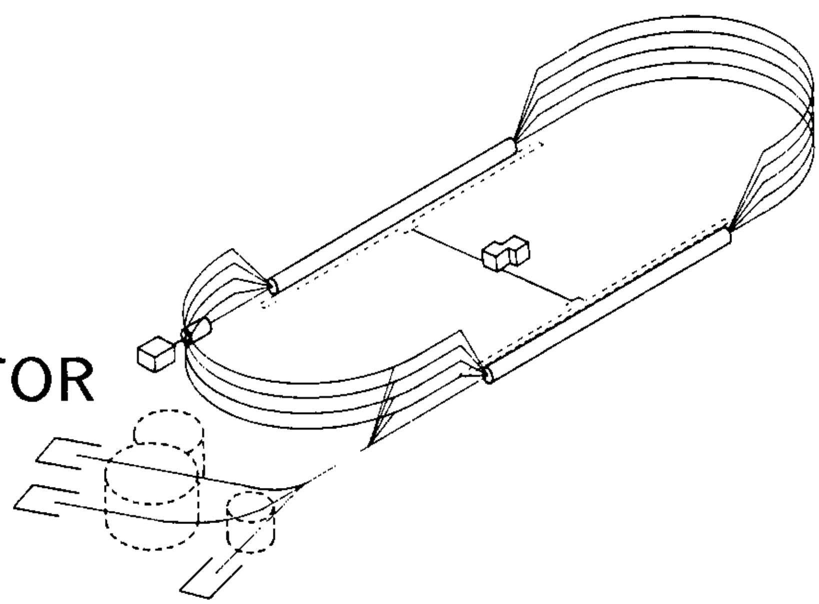

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The Southeastern Universities Research Association (SURA) operates the Continuous Electron Beam Accelerator Facility for the United States Department of Energy under contract DE-AC05-84ER40150.

Solvents and Pumpdown Characteristics of SRF Nb Cavities*

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Abstract

High-performance superconducting radio frequency (SRF) cavities are placed under UHV and low particulate conditions when they go through the surface treatment process, and are kept under these conditions permanently thereafter, both before and during use in the accelerator. The outgassing of the gasket material in the gate valves and the outgassing of the solvents that come in contact with the gasket and the cavity surfaces determine the ultimate pressure that can be obtained in the cavity. A systematic study of gaskets and various solvents was undertaken in order to understand the conditions that lead to a low ultimate pressure in a short period of time. The results with Kalrez, Viton O-rings and solvents like methanol, acetone, isopropyl and freon will be presented in this paper. Also, we discuss the effect of a slow or fast initial pumpdown in order to prevent the settlement of particulates due to condensation.

I. INTRODUCTION

Superconducting accelerator cavities made from high-purity niobium are presently limited in their high-gradient performance by either field emission loading or thermal-magnetic breakdown at microscopic defects. It is generally accepted that field emission is either caused by artificial contamination of the sensitive cavity surfaces due to handling and assembly procedures or by intrinsic emitters embedded in the surface. There are in addition indications that the condensation of residual gases in the evacuated cavities can contribute to field emission loading.

Several precautions have been taken at CEBAF to minimize the potential for artificial contamination [1]: CEBAF's basic building block—the cavity pair—consists of two 5-cell 1500 MHz cavities, which are hermetically sealed when assembled in a class 100 clean room after chemical surface treatment and final solvent rinsing.

During this assembly procedure two ceramic rf windows are mounted onto the fundamental power coupling waveguide, the beam-pipe ends of the cavity pair are closed off with high-vacuum O-ring sealed gate valves and the higher-order-mode couplers are attached to the HOM-waveguide openings. The outgassing of all these components and in particular the outgassing of the O-ring materials that come in contact with solvent vapours or solvents used for final rinsing of the cavities and the cavity surfaces themselves determine the ultimate pressure attainable in the cavity pair.

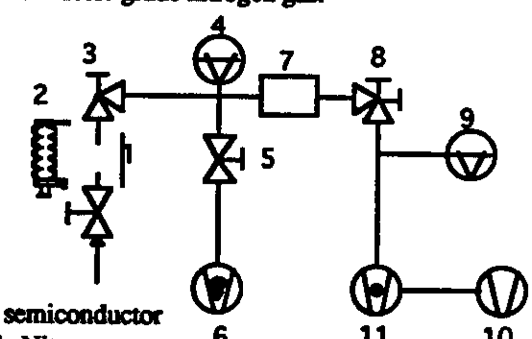
Results of a systematic study [2] of the outgassing characteristics of O-ring materials such as Kalrez and Viton and solvents such as methanol, acetone, isopropyl and freon are reported in the following sections.

In addition we have investigated the influence of the solvents, viz. methanol and isopropyl, on the removal of water from the 5-cell Nb cavity. Further, the effect of pumpdown speed during the initial evacuation process of the cavities on particulate generation (which is well known in the semiconductor industry) is also studied.

II. EXPERIMENTAL SETUP

A. Outgassing Experiments

For the pumpdown characteristics studies a small UHV system as shown schematically in Figure 1 was assembled. A 50 l/sec turbomolecular pump was used for a short time for rough pumping the UHV system, consisting of a 60 l/sec star cell ion pump (Varian), a residual gas analyzer (RGA, Hiden) and a standard UHV nipple as the vacuum chamber holding the O-ring. At the beginning of each test, the UHV system was baked at 425 K for 12 hours to reach an ultimate pressure of $p \approx 2 \cdot 10^{-10}$ torr as indicated by the RGA with the dominant residual gas being H_2. Prior to loading the chamber with the O-ring material, it was vented with dry 99.999% semiconductor grade nitrogen gas.



1. Nipple 2. Cavity 3, 5. All metal valves 4. RGA
6. Ion pump 7. Particle counter 8. Two stage valve
9. Ion gauge 10. Rotary pump 11. Turbomolecular pump
Figure 1. Schematic of experimental setup.

B. Cavity Pumpdown and Particle Generation Experiments

For this type of experiments the same arrangement as shown in Figure 1 was used with the exception that a 5-cell cavity was the vacuum chamber and a 170 l/sec turbomolecular pump replaced the smaller pump.

* This work was supported by DOE contract DE-AC05-84ER40150.

In the particle generation experiments the pumping speed was regulated by a two-stage valve (HPS) consisting of a small by-pass valve made out of an aperture and a regular valve. A commercial particle detection system (TSI, Inc.) was placed between the cavity and the pump to monitor the amount of particles generated during evacuation.

III. RESULTS AND DISCUSSION

A. Kalrez and Viton O-Rings

A virgin Kalrez O-ring was placed in the nipple and the UHV system was pumped down to $2\cdot10^{-5}$ torr with the turbo pump, and the getter-ion pump was turned on after isolating the turbopump. On reaching a pressure of $1\cdot10^{-9}$ torr, the O-ring was removed from the nipple and soaked in methanol for 5 minutes. The pumpdown procedure was repeated with the methanol-soaked O-ring. The same procedure was also adapted with the Viton O-ring. Figure 2 shows the pumpdown characteristics of the virgin and methanol-soaked O-rings. The same ultimate pressure was obtained with both the Kalrez and Viton O-rings after 50 hours of pumpdown. The residual spectra for each O-ring indicate that besides H_2, fluorine is the major residual component for Kalrez, and water in the case of Viton.

The methanol soaked O-rings were not baked during the first 50 hours of pumpdown except for one minute of hot air exposure onto the nipple. A final pressure of $1\cdot10^{-9}$ torr was reached with the Kalrez O-ring in 50 hours, whereas the Viton O-ring needed to be baked for 2 hours with 425 K hot air exposure onto the nipple to reach an ultimate pressure of $2\cdot10^{-9}$ torr in 175 hours. The RGA spectra indicate that methanol is the major residual gas component besides hydrogen with both of the O-rings. Fluorine is still the dominant residual gas in the case of Kalrez and water in case of the Viton. Since surface areas of both the O-rings are similar (~30 cm^2), one can directly compare their pumpdown characteristics. It appears that the Viton O-ring absorbs larger amounts of methanol and it needs to be baked out to desorb methanol. So the pumpdown characteristics of Kalrez are superior to those of Viton gasket material.

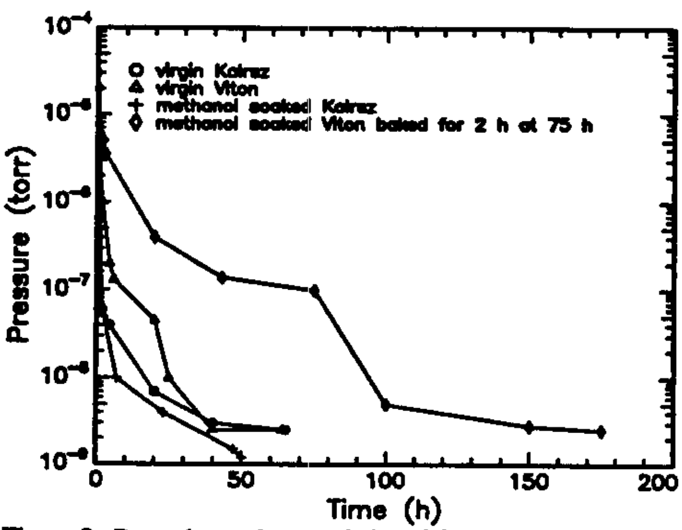


Figure 2. Pumpdown characteristics of O-rings.

A similar procedure was carried out with ethanol-soaked Kalrez and Viton O-rings, but the pressure in the system remained at $1\cdot10^{-8}$ torr even after pumping for a month. Similar results were obtained with freon-soaked O-rings. It appeared at this time that the slow outgassing of ethanol and freon themselves may be the reason for such results. So it was decided to carry out the pumpdown characteristics of the solvents in the absence of any gasket material.

B. Solvents

As usual, the UHV system was baked to reach a pressure of $2\cdot10^{-10}$ torr and the nipple was vented with the dry nitrogen. The inner surface of the nipple was wiped with a clean cloth wetted by the solvent under investigation. The pumpdown followed the same procedure as described above. Figure 3 shows the pumpdown characteristics of the solvents methanol, ethanol, isopropyl alcohol, acetone and freon. As can be seen the pumpdown times for methanol and acetone are much shorter to reach a pressure of $2\cdot10^{-9}$ torr compared to ethanol, isopropyl alcohol and freon. In fact, it is very difficult to reach ultimate pressures of less than 10^{-8} torr with ethanol, isopropyl alcohol and freon with out a proper bake-out.

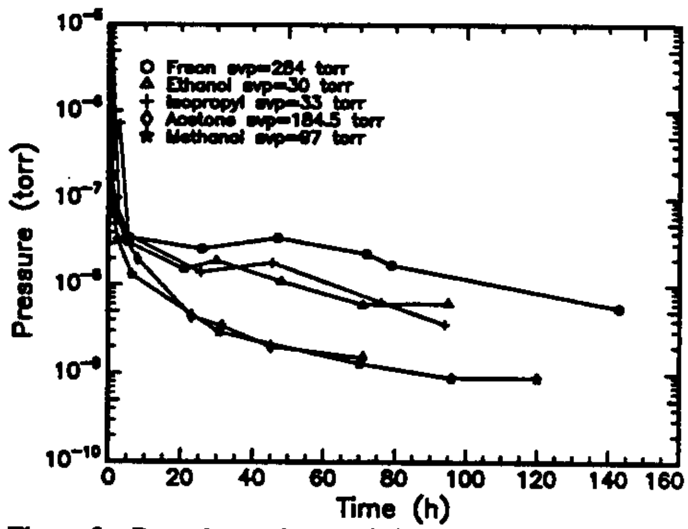


Figure 3. Pumpdown characteristics of solvents [saturated vapor pressure at 20°C=svp].

C. Cavity Pumpdown

At CEBAF a 5-cell Nb cavity is first rinsed with ultrapure water after the chemical surface treatment followed by a twofold solvent rinse with methanol or isopropyl alcohol to remove the water from the surface. Two cavities are assembled into a pair with all the auxiliary components and pumped down with a turbo pump for a period of 15 minutes; then further pumping is done with ion pumps. This procedure was adopted to minimize the contamination of the cavity surfaces with hydrocarbons from the pumping system. Figure 4 shows the partial pressure (PP) of water in a 5-cell cavity as a function of pumpdown time with the turbo pump and ion pump systems for methanol and isopropyl alcohol solvent rinses. The turbo pump system reduces the PP of water to the 10^{-8} torr range within a period of 7.3 hours. The PP of water in the methanol-rinsed cavity is lower than the isopropyl-rinsed cavity. In the case of the ion pump pumped cavity the pressure never improves to the 10^{-8} torr range even after pumping for more than 13 hours. Again the PP of water is higher in the case of the isopropyl-rinsed cavity.

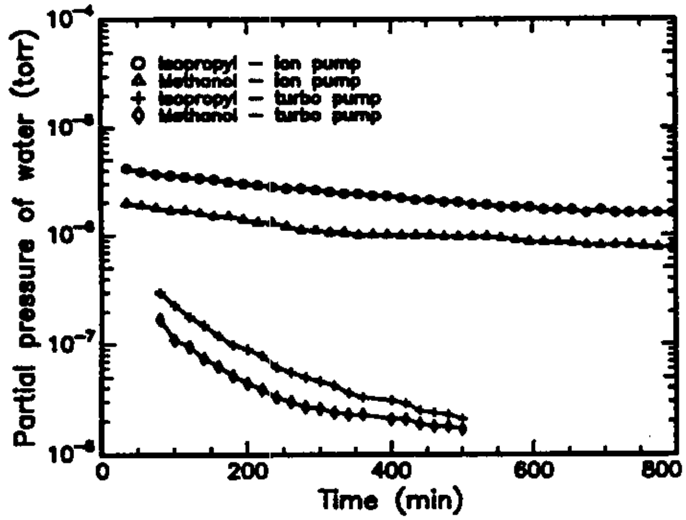


Figure 4. Partial pressure of water in a 5-cell cavity.

D: Particle Generation

The artificial contamination of the sensitive cavity surfaces during the handling and assembly procedures is recognized as one of the mechanisms responsible for the field emission loading of the cavities, despite precautions like assembly in a class 100 clean room and the use of high-purity chemicals and solvents.

Entrainment of residual particulates from condensation of solvents has been identified as the major source of contamination in the silicon wafer processing technology [3,4] mainly originating from particulate-laden process vacuum chambers and load locks.

Since the cavity surfaces are chemically cleaned and rinsed with very clean solvents, the reentrainment of particulates is drastically reduced in comparison. Furthermore the cavities have low relative humidity conditions due to the rinsing with solvents, and the formation of liquid particles due to fast initial evacuation is reduced.

Measurements have been done to verify these assumptions: the number of particles measured during the unthrottled pumpdowns is less than 4 per 25 liters of solvent vapour removed from the cavity pair. Further, this fast (2 minutes to reach 10^{-2} torr from atmospheric pressure) removal of solvent from the cavity surfaces is expected to drag away the remaining water molecules from the surface, leaving a drier surface behind. Slow (20 minutes to reach 10^{-2} torr from atmospheric pressure) pumpdowns through the orifice appear to have produced more particles due to the expansion and also seem to have inhibited the removal of water from the cavity surfaces in a short period.

IV. CONCLUSIONS

In the absence of baking, solvent-soaked Viton O-rings are prolonging the pumpdown time to reach lower pressures in comparison to Kalrez O-rings. In addition, pumpdown times are much shorter for methanol and acetone compared to ethanol, isopropyl alcohol and freon for achieving low pressures. Methanol removes water more effectively from the cavity surfaces in comparison to isopropyl alcohol, thereby facilitating better cavity pumpdowns with ion pumps to lower pressures. Neither slow nor fast pumpdown of the cavities contributes to particulate reentrainment or particle generation in cavity pairs. However, fast pumpdown is likely to remove more water from cavity surfaces.

V. ACKNOWLEDGMENTS

We would like to thank J. Mammosser and K. Yopp for their help with cavity rinsing and the drawing of the sketch respectively.

VI. REFERENCES

[1] P. Kneisel *et al.*, "Superconducting Cavities from High Thermal Conductivity Niobium for CEBAF," Proc. of the Conf. on Electron Beam Melting and Refining, Reno 177 (1990) Bakish Materials Corporation .

[2] M. G. Rao, "Outgassing of Kalrez, Viton O-rings and Solvents," CEBAF Report TN0185 (1989.)

[3] John F. O'Hanlon, "Advances in Vacuum Contamination Control for Electronic Materials Processing," *J. Vac. Sci. Technol.* A5, 2067 (1987).

[4] Degang Chen *et al.*, "Vacuum Particle Generation and Nucleation Phenomena During Pumpdown," *J. Vac. Sci. Technol*. A8, 933 (1990).